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LEACH TEST OF SIX 192-IRIDIUM PELLETS BASED ON THE IAEA "SPECIAL FORM" TEST PROCEDURES

by

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LEACH TEST OF SIX 192-IRIDIUM PELLETS BASED ON THE IAEA "SPECIAL FORM" TEST PROCEDURES

ABSTRACT

The designation "special form" may be applied to indispersible solid radioactive material if the material meets the requirements of the tests described in the International Atomic Energy Agency Regulations for the Safe Transport of Radioactive Type Materials, 1973 Revised Edition, Safety Series No. 6*.

One of the tests, the leach assessment, which is required to be carried out following specified mechanical tests, requires that specimens be immersed for seven days in a water bath at ambient temperature, then the bath heated to 50°C for 4 hours. The specimens are then placed in an atmosphere of still air with 90% humidity at 30°C for seven days, followed by being immersed in a 50°C water bath for 4 hours. The activity of each water bath, following the test, must not exceed 0.05 µCi (1.85 x 10^3 Bq).

This report presents the procedures and results of a leach test performed on 6 "as received" active 192-iridium pellets. Mechanical tests were not carried out prior to or following the leach test. The activity of each of the first 6 water baths was found to be between 2.3 and 12.6 µCi, (8.5 x 10^4 and 4.7 x 10^5 Bq) and of the second 6, between 1.3 to 6.0 µCi, (4.8 x 10^4 to 2.2 x 10^5 Bq) thus exceeding the allowable limit.

RÉSUMÉ

On attribue le terme "forme spéciale" à une matière radioactive solide non susceptible de dispersion qui satisfait aux exigences des épreuves stipulées dans le Règlement de transport des matières radioactives de l'Agence internationale de l'énergie atomique, Collection "Sécurité" n° 6, édition révisée de 1973.*

Parmi ces épreuves, l'évaluation des effets de la lixiviation qu'il faut effectuer à la suite d'épreuves mécaniques précises, exige l'immersion des échantillons dans l'eau pendant sept jours à la température ambiante, et ensuite pendant quatre heures dans la même eau chauffée à 50° C. Les échantillons sont alors conservés, pendant sept jours, dans de l'air immobile, à 90% d'humidité et à 30° C, puis immergés de nouveau, pendant quatre heures, dans de l'eau à 50° C. À chaque stade de l'épreuve, l'activité de l'eau ne doit pas dépasser 0,05 µCi (1,85 x 10^3 Bq).

Le présent rapport présente les étapes et les résultats d'une épreuve de lixiviation effectuée sur six pastilles non modifiées d'iridium 192 actif. Aucune épreuve mécanique n'a été effectuée avant ou après l'épreuve de lixiviation. L'activité de chacun des six premiers bains d'eau a varié entre 2,3 et 12,6 µCi (8,5 x 10^4 et 4,7 x 10^5 Bq) et celle de chacun des six derniers bains, entre 1,3 et 6,0 µCi (4,8 x 10^4 et 2,2 x 10^5 Bq), soit plus que la limite prescrite.

*This edition of the regulations does not quote activity limits in SI-units; therefore, in order to maintain consistency, old units are preferred in this report, and SI-units are shown in brackets.

Cette édition du Règlement ne présente pas les limites d'activité en unités SI; aux fins d'uniformité, l'auteur du présent rapport utilise les anciennes unités et donne les équivalents SI entre parenthèses.

DISCLAIMER

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1. INTRODUCTION

The International Atomic Energy Agency, (IAEA)* defines special form radioactive material as follows:
"Special form radioactive material shall mean either an indispersible solid radioactive material or a sealed capsule shall be so constructed that it can be opened only by destroying the capsule. Special form radioactive material shall meet the following requirements:

a) It shall have at least one dimension not less than 5 mm.

b) It shall comply with the relevant test requirements in section VII, paras. 726-737.

The special form concept permits the inclusion of a greater activity in a Type A package".

The relevant tests are the impact test, percussion test, bend test, heat test, and leach test; details of the test requirements are given in Appendix 3 herein.

This report investigates the tests within the terms of a contract, let to Monserco by the Department of Supply and Services Canada (DSS). The scope of work is specified in Appendix A of the contract, and modified by a DSS telex dated December 8, 1980. The intent of the modification to the scope of work is to place the emphasis on and to carry out an actual leach test. Therefore, this report presents the procedures and results of a leach test performed on six activiated 192-iridium (192-Ir) pellets, supplied by Atomic Energy Canada Limited, Radiochemical Company (AECL-RCC) and addresses to a limited extent, the remaining required mechanical tests.

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* IAEA Regulations for the Safe Transport of Radioactive Materials, 1973, Revised Edition. Safety Series No. 6.

Since the leach test was performed without first carrying out the mechanical tests, it does not comply with the chronological testing order specified in IAEA Regulations. The test does however permit a comparison of the behaviour of as-received material with the levels of activity allowed by the regulations.

For example, it would normally be expected that tests carried out on as-received material would show activity levels lower than tests carried out on material that had first suffered mechanical damage. However, as will be seen in this report, the results indicate activity levels above the allowable thereby demonstrating the importance of the leach test in a practical case.

The pellets used for the test were selected on the basis of availability and do not meet the geometric requirements of the IAEA regulations; however, they are entirely suitable for purposes of the leaching assessment.

The intent of the IAEA regulations is to limit the activity release from special form indispersible solid or encapsulated material, under accident conditions. The leach test is therefore designed such that only the total amount of activity released or leached from the material is required to be measured. Consequently leach rates, solubility, diffusion rates or similar mass transfer parameters were not considered.

2. PELLET SPECIFICATIONS

2.1 Activity

Six 192-iridium pellets, each with an activity of 26.8 Ci (992 GBq at the time of shipment March 25, 1981 from AECL-RCC were used in the test. The half-life of 192-Ir is approximately 74 days.

2.2 Exposure

The average gamma energy per disintegration of 192-Ir is about 0.5 MeV[1]. The exposure rate at one meter of an 192-Ir source is about 0.48 R/h-Ci[1]. 192-Ir decays into stable 192-Pt by the emission of a β particle of a maximum energy of approximately 0.67 MeV[1].

2.3 Size

Each pellet was made from solid iridium metal, nominally 3 mm diameter by 0.5 mm thick. Six inactive pellets were measured; the dimensions are given in the following table.

PELLETS	THICKNESS (mm)	THICKNESS (Inches)	DIAMETER (mm)	DIAMETER (Inches)
1	0.533	(0.021)	3.05	(0.120)
2	0.610	(0.024)	2.97	(0.117)
3	0.559	(0.022)	3.00	(0.118)
4	0.483	(0.019)	3.00	(0.118)
5	0.533	(0.021)	3.00	(0.118)
6	0.559	(0.022)	3.00	(0.118)

(NSIL Micrometer YUAAOZ 1")

2.4 Composition

The mass of each pellet is nominally 0.08 g, 99.5 to 99.8% iridium.

2.5 Production

The pellets were produced by neutron irradiation of inactive 191-Ir, whose total thermal neutron activation cross-section (isometric + ground state) is approximately equal to 956 barns (1 barn = 10^{-24} cm^2)[1]. They were irradiated at Chalk River Nuclear Laboratories (AECL-Research Company), and supplied by AECL-Radiochemical Company.

2.6 Packaging

The pellets were shipped by AECL-RCC in a F113 B container. The contents are described below.

The pellets were contained in a screw-top hexagonal cross-section, aluminum capsule, 3.8 cm high, 1.6 cm across the flats. This capsule was contained inside a cylindrical screw-top brass capsule, 4.4 cm high by 2.8 cm diameter. The lower section of the brass

capsule has a hexagonal cross-section. The screw-top is made from steel and has a flat projection with a hole to permit it to be unscrewed and removed using a simple bent wire lifting device.

The brass capsule was contained inside a cylindrical steel/lead flask 23 cm high and 18 cm diameter. The flask lid is attached by four bolts and may be lifted by means of a lifting eye.

The flask was transported inside a steel drum approximately 51 cm high by 42 cm diameter. The space between the steel flask and the drum was filled with wooden spacers. The lid was secured by a metal band.

2.7 General Properties of Iridium

Comprehensive information on the properties of iridium was difficult to locate. A brief literature survey (see Appendix 2) provided some data. Englehard Industries, the supplier of iridium to AECL, were unable to provide any useful data.

3. LEACH TEST PROCEDURES

Procedures for carrying out the leach test were developed and later modified following a rehearsal with inactive pellets. The procedures are described in detail in Appendix 1 and briefly as follows.

3.1 General Procedures

The test was carried out in the reactor hot cell at McMaster University. The cell is a room about 2.1 x 3.7 meters with walls about one meter thick and a square lead glass window about 61/cm side by one meter thick.

There was one set of manipulators and a number of removable shield plugs to permit cables to be led into the cell. In order to minimize the risk of dropping a pellet beyond the reach of the manipulators, a three sided wooden box, fitted with a remotely operated device for lifting the flask lid, was positioned against the lead glass window. All operations were conducted within the confines of the box, all corners of which could be seen through the hot cell window and accessed by the manipulators. Inside the box was positioned the dry bath heaters, test tube racks, fixtures for aiding the removal of the tops from the capsules in which the pellets were shipped, the pierced ladles in which the pellets were placed during the test and other items used for manipulating the capsules and pellets.

The flask, in which the pellets and intermediate capsules were shipped, excluding the steel drum and wooden spacers, was positioned in the back of the box. The lid was lifted, after removing the 4 bolts, by means of a small block and tackle arrangement operated from outside the hot cell through one of the shield plug holes. A mirror attached to the box allowed a view of the inside of the flask and the brass capsule with its steel screw-top was lifted out of the flask using a magnet on the end of a wire; the wire was gripped by one of the manipulators.

The brass capsule was placed in a fixture with a hexagonal hole; this gripped the capsule while the top was unscrewed using one of the manipulators.

The brass capsule was then picked up and the aluminum capsule tipped out. The aluminum capsule was placed

in a fixture similar to that used for the brass capsule and the screw top removed using the manipulators. The aluminum capsule was then lifted and the 6 pellets carefully tipped out.

The pellets were placed one at a time into the ladles and the ladles placed in the first set of test tubes labelled A1 to A6. The test tubes had sufficient water to cover the ladles. The pellets were lifted and placed in the ladles using a small suction probe. The suction was provided by a vacuum pump placed in the hot cell and controlled from outside the cell; a remote valve was used to bleed air into the suction line to release the pellet once it was in position over the mouth of the ladle. The suction probe was connected to the vacuum pump through a filter device. The pump exhausted into the hot cell. Once the six pellets were placed in the test tubes, the test commenced.

At the end of 7 days, test tubes "A" containing the pellets were heated to 50°C and held for 4 hours. Following this, the ladles, with the pellets, were transferred to the humidity chambers (test tubes "B1 to B6") and allowed sit for 7.5 days. The temperature was maintained at 30°C during this period by the second dry bath heater.

The first set of test tubes (A1 to A6) was transferred to the test tube rack and a third set, labelled C1 to C6, containing sufficient water to cover the ladles and pellets was placed in the first dry bath heater. Following the 7.5 days in the humidity chambers, the ladles, containing the pellets were placed in the third set of test tubes for 4 hours, the water having been heated to 50°C prior to transferring the ladles.

The ladles were then removed one at a time from the 50°C water bath and the pellets tipped out onto absorbent paper. Some difficulty was experienced removing the pellets from the ladles due to the pellets having adhered to the ladles, caused by what is believed to be the result of beta decay heat and the ladles being made from plastic; the pellets appear to have gotten sufficiently hot that they locally softened the plastic causing an indentation in which the pellets adhered.

The pellets were then placed in the aluminum capsule and the top screwed on, the aluminum capsule then placed in the brass capsule and the top screwed on, the brass capsule placed in the shipping flask and the flask lid placed in position ready for bolting down.

The hot cell was then opened and radiation levels monitored before entering to retrieve the test materials and equipment. Swipe tests were carried out before removing any equipment. Certain items of the equipment were found to be contaminated and were disposed of as radioactive waste.

Measurements were then carried out on the test tubes with contents intact, as well as on the ladles. The results are summarized in section 5 of this report.

3.2 Temperature Control

The IAEA regulations specify certain temperatures for each step of the leach test. The temperatures were controlled as described below.

a) Water at Ambient Temperature (IAEA Step a)

The pellets were placed in test tubes, with water at ambient temperature, in the dry bath

heater, but the heater was not turned on. The temperature of the heater was measured each day during the 7 day testing (see Table 1, Appendix 1). This temperature represents the ambient temperature in the hot cell.

b) Water at 50°C (±5°C) (IAEA Steps b and e)

The heater used to raise the water temperature to 50°C (±5°C) was tested at Monserco's Laboratory for four consecutive days. It was also turned on and off many times, both from cold and warm initial conditions and found to be always operable. The temperature of the heater block was measured with a mercury thermometer and a thermocouple. The heater fulfilled the specifications and heated up to the desired temperature in about 30 minutes.

Test tubes with the same amount of water used in the actual test were placed in the heater block. A ladle with an inactive pellet was placed in each tube. The heater control was adjusted to give a water temperature of 50°C, (the heater has an automatic shut-off device to maintain the set value). The temperature of the water was measured with a mercury thermometer and the temperature of the heat block measured using a thermocouple. A block temperature of 62° was found coverhand to a water temperature of 50°C. Just prior to the leach test, the heater was checked to ensure repeatability inside the hot cell.

c) Temperature at 30°C (IAEA Step d)

A second heater was similarily tested and calibrated to maintain the air in the humidity chambers at 30°C. The humid air temperature was measured with the mercury thermomter. A block temperature of 42°C was found to correspond to humid air temperature of 30°C. Just prior to the leach test the heater was tested again inside the hot cell.

3.3 pH Control (IAEA Step a)

The water used for the test complied with the IAEA specified pH range of 6-8. Reactor-quality water was used in the test; prior to the initiation of the test, the measured pH was 7.1 (see Appendix 1).

3.4 Conductivity Control (IAEA Step a)

The reactor-quality water used for the test surpassed the IAEA requirements of a maximum conductivity of 10 µmho at 20°C. The measured conductivity was 1.7 µmhos.

The ladles were made of polyethylene in order to avoid metal impurities during the test, and stoppers on the test tubes prevented dust from falling into the tubes affecting the pH and conductivity.

3.5 Humidity Control

The second stage of the test requires that the specimens be stored for seven days in still air of humidity not less than 90% and at 30°C. Each specimen was suspended in a test tube, about 1/3 full of water, such as to be just above the level of the water. The test tubes were closed using rubber stoppers.

The humidity was not measured; however, calculations were carried out which indicated that the humidity in the test tube would reach 90% within about 5 minutes after being stoppered. The calculations, assuming only molecular diffusion of water vapour are as follows:

The rate of change of humidity with respect to time in a one dimensional system is given by:

$$\frac{dH}{dt} = \frac{kd^2H}{dx^2}$$

where H is the percentage humidity, x the length of the test tube above the water level, and k is the diffusivity of water vapour in air. The boundary conditions assumed are that at the surface of the water the humidity is 100%, and at the closed end the rate of change of humidity with respect to distance (i.e. humidity gradient) is zero. From reference 2 the diffusivity of water in air is 0.260 cm^2/sec. at 25°c.

Solving the above equation using finite difference techniques for length of 9 cm gives the results shown in Table 1. It can be seen that after 300 secs the humidity at any point is greater than 90%.

4. EQUIPMENT

The equipment used in the test is listed in Appendix 4.

5. RESULTS

5.1 Activity Measurements

Detailed results of the leach test are presented in Appendix 1. Summarizing, the test tubes in which the pellets were placed for the 7 days ambient temperature immersion test contained between 2.3 and 12.6 µCi (8.5×10^3 and 4.7×10^5 Bq).

The final water immersion test tubes contained 1.3 to 6.0 μCi (4.8×10^3 to 2.2×10^5 Bq), and the ladles, 2.5 to 24.6 μCi (9.3×10^4 to 9.1×10^5 Bq) Although not required, the water in the humidity chambers was also measured, and found to be between 0.01 and 0.08 μCi (3.7×10^2 and 3.0×10^3 Bq).

5.2 Discussion of Results

The leach assessment was carried out in accordance with the IAEA Regulations for The Safe Transport of Radioactive Materials 1973 Revised Edition, Section VII, para 736 (see Appendix 3). In carrying out the test, the pellets were in the "as normally shipped" condition, and it is most likely that they would have loose surface particles, or iridium dust loosely adhered to the surface that would easily be washed off mainly during the first part of the test. The test as performed is not therefore a leach test where only intergranular attack by the solvent occurs thus liberating molecules of iridium. The test does meet the intent of the IAEA regulations to the extent of limiting an activity release whether by moleular leaching or the washing off of surface contamination. However, if the pellets had been washed prior to shipping, the results may have been substantially different.

6. CORRECTION FOR DECAY

In some instances, it may be desireable to correct for decay. Let A_1 be measured activity resulting from the test, The leached activity A_0 relative to the time of shipment will be:

$$A_0 = \frac{A_1}{e^{-\lambda t}}$$

where λ (s^{-1}), the decay constant of 192-Ir, is equal to 1.08×10^{-7}; and t (s) is the time span between shipment

and the test measurement. Since the activity levels measured in the water exceeded the allowable limit of .05 μCi, correction for decay was not required; the correction is only appropriate when the measured activity is less than the allowable.

7. MECHANICAL TESTS

In addition to the leach test, special form material is required to withstand the following mechanical tests:

i) Impact Test
ii) Percussion Test
iii) Bend Test
iv) Heat Test

7.1 Impact and Percussion Test

The impact test requires that the specimen be allowed to fall freely a distance of 9m onto a specified target. The percussion test involves dropping a billet onto the specimen which is resting on a specified anvil. The bend test is applicable only to specimens 10 cm long or longer whose aspect ratio is 10:1 or greater. Because the specimens which are the subject of this report are not within these geometry limits, the bend test will not be addressed.

The specimen must not break or shatter when subjected to the mechanical tests. The term break or shatter is somewhat nebulous since there is no quantitative limits given in the IAEA Regulations for breaking or shattering. Breaking or shattering into pieces that are easily discernable by the naked eye is quite different from breaking off of microscopic particles. The question then becomes how can one decide whether or not breaking or shattering has occurred if there is no apparent damage.

In the case of the leach assessment, excess leaching is deemed to have occurred if the activity released from the specimen is greater than 0.05 μCi (1.85×10^3 Bq). The specific activity of the specimens leach tested is,

$$\frac{26.8}{\frac{\pi \times 3^2 \times 0.5}{4}} = 7.58 \text{ Ci/mm}^3$$

$$= 2.80 \times 10^2 \text{ GBq/mm}^3$$

Assume the activity to be uniformly distributed within the specimen, an activity release of .05 μCi would occupy a volume of $.05 \times 10^{-6}/7.58 = 0.66 \times 10^{-8}$ mm³. Assuming that a single particle in the shape of a cube broke off the specimen it would have sides 0.002 mm or a spheroid would have a diameter of $2(\frac{3}{4\pi} 0.66 \times 10^{-8})^{1/3} \approx .002$ mm. To detect or locate a particle of this size even inside an enclosure of the same order of magnitude as the pellet would be difficult. In addition, the pellet must be thoroughly washed prior to the test to ensure that the shattered particles were the result of the mechanical test.

One approach would be to swipe the target and and billet areas and the swipes measured to a detectable limit of less than 0.05 μCi. If the swipe tests indicate less than 0.05 μCi relative to the original activity level at the time of shipment, it would be concluded that no physical damage had occurred, i.e. the specimen did not break or shatter, thereby meeting the intent of the IAEA regulations. After each mechanical test is completed, the specimen would be subjected to a leach test in water, as described in section 3.

7.2 Heat Test

The heat test involves heating the specimen to 800°C for 10 minutes then allowing it to cool. The specimen must not melt or otherwise disperse. Inactive pellets are almost pure iridium (more than 99%). Iridium melts at about 2450°C therefore the pellets would comply with

the IAEA melting requirement at 800°C. Platinum also melts at temperatures higher than 800°C, so decayed pellets of Ir/Pt composition also comply with the intent of the IAEA requirement, except perhaps, for a very small fraction (if any at all) of eutectic alloy that may be formed.

Iridium forms a volatile oxide at temperatures of about 1000°C. Hence, the pellets also comply with the intent of the IAEA dispersion requirement at 800°C.

However, to comply with the IAEA regulations the heat test followed by the leach test should be performed since it is not known if there would be any physical damage due to heating. For example, at 800°C there could be oxidation and scaling of the surface that could affect the leach assessment.

8. CONCLUSIONS

The leach test was carried out successfully with regard to being able to follow with sufficient accuracy, the test procedures specified in the IAEA Regulations. The test results indicate that the "as shipped" specimens would not comply with the IAEA allowable activity release even without first carrying out mechanical testing. It was not possible to determine whether or not the activity release was a result of initial contamination or leaching so that the activity release may depend more on decontamination prior to shipping, than leaching.

TABLE 1.

Humidity v.s. Time and Distance

Time Seconds	H_2O Surface	% Humidity								Stopper
.5	100.0	13.0	1.7	.2	.0	.0	.0	.0	.0	.0
1.0	100.0	22.8	4.2	.7	.1	.0	.0	.0	.0	.0
1.5	100.0	30.5	7.2	1.5	.3	.0	.0	.0	.0	.0
2.0	100.0	36.5	10.3	2.5	.5	.1	.0	.0	.0	.0
2.5	100.0	41.3	13.3	3.6	.9	.2	.0	.0	.0	.0
3.0	100.0	45.3	16.2	4.9	1.3	.3	.1	.0	.0	.0
3.5	100.0	48.6	18.9	6.3	1.8	.5	.1	.0	.0	.0
4.0	100.0	51.4	21.5	7.7	2.4	.7	.2	.0	.0	.0
4.5	100.0	53.9	23.9	9.1	3.1	.9	.3	.1	.0	.0
5.0	100.0	56.0	26.2	10.5	3.8	1.2	.4	.1	.0	.0
5.5	100.0	57.8	28.2	12.0	4.5	1.5	.5	.1	.0	.0
6.0	100.0	59.5	30.2	13.4	5.3	1.9	.6	.2	.1	.0
6.5	100.0	60.9	32.0	14.7	6.0	2.3	.8	.2	.1	.0
7.0	100.0	62.2	33.7	16.1	6.9	2.7	.9	.3	.1	.1
7.5	100.0	63.4	35.3	17.4	7.7	3.1	1.1	.4	.1	.1
8.0	100.0	64.5	36.7	18.6	8.5	3.5	1.4	.5	.2	.1
8.5	100.0	65.5	38.1	19.8	9.3	4.0	1.6	.6	.2	.1
9.0	100.0	66.4	39.4	21.0	10.2	4.5	1.8	.7	.3	.2
9.5	100.0	67.3	40.7	22.2	11.0	5.0	2.1	.8	.3	.2
10.0	100.0	68.1	41.8	23.3	11.8	5.5	2.4	1.0	.4	.3
20.0	100.0	77.2	56.5	39.1	25.7	16.0	9.5	5.6	3.5	2.9
30.0	100.0	81.3	63.8	48.2	35.2	24.9	17.2	12.0	9.0	8.2
40.0	100.0	83.8	68.4	54.4	42.2	32.2	24.4	18.9	15.6	14.7
N										
50.0	100.0	85.6	71.8	59.1	47.9	38.4	30.9	25.6	22.4	21.4
60.0	100.0	87.0	74.6	63.0	52.7	43.9	36.9	31.9	28.9	28.0
70.0	100.0	88.2	76.9	66.4	56.9	48.9	42.4	37.7	34.9	34.1
100.0	100.0	91.1	82.5	74.5	67.3	61.1	56.1	52.5	50.1	49.7
150.0	100.0	94.4	88.9	83.8	79.2	75.3	72.1	69.8	68.5	68.1
200.0	100.0	96.4	93.0	89.7	86.8	84.3	82.3	80.9	80.0	79.7
250.0	100.0	97.7	95.5	93.5	91.6	90.0	88.8	87.9	87.3	87.1
300.0	100.0	98.6	97.2	95.9	94.7	93.7	92.9	92.3	91.9	91.8

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APPENDIX 1

TEST RESULTS

^{192}Ir LEACH TEST REPORT

FOR

MONSERCO LIMITED

By

P.C. ERNST - MNR

APRIL 23, 1981

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A. INTRODUCTION

This report covers the leach tests carried out on six iridium pellets in the Hot Cell at the McMaster Nuclear Reactor during April, 1981. The procedures followed and measurements made were as directed by Monserco Limited. All equipment and test apparatus used in the actual experiment was either supplied by Monserco or as requested by Monserco.

The test is reported on in five sections; preparations, actual test run, clean-up, subsequent determination of activities in the test tubes and ladles, and the overall conclusions reached.

B. TEST RUN PREPARATIONS

The iridium pellets arrived at MNR at 1600 hours on Friday, March 27, 1981. The test run was then scheduled to commence on April 1, 1981.

B.1 Water for Baths

On April 1, 1981 a mixture of demineralized pool make-up water and water passed through an Anion exchange column was prepared with the following measured characteristics.

pH: 7.1

Conductivity: 1.67 μmhos

The pH was measured with a Beckman Zeromatic SS-3 pH meter. The conductivity was measured via an Industrial Instruments Conductivity Bridge Model RC16B1. The initial conductivity measurement at 20°C was 670,000 ohm-cm which, when corrected for temperature, yielded a true conductivity of 6.0×10^5 ohm-cm or 1.67 μmhos.

B.2 Introduction of Iridium Castle to Hot Cell

At 1400 hours on April 1, 1981, AECL shipping container F113-20 was unsealed and opened. The external radiation levels measured were 30 mr/hr at contact and 2.5 mr/hr at 1 m. The shipping container was checked outside and inside for contamination. All accessible surfaces were clean.

The inner castle, containing the iridium, was then removed and moved into position on the experimental table in the Hot Cell. Radiation levels were 100 mr/hr at contact with the castle. The lid bolts were then removed and set aside.

Before closing up the Hot Cell, the manipulator fingers were wrapped in clear Mactac and then inserted into fingers cut from a plastic glove. The Hot Cell door was then closed.

C. TEST RUN

C.1 Loading Pellets into Group A Test Tubes

With the Hot Cell secured, the lid was removed from the iridium castle via the remotely operated block and tackle arrangement. The lid was set down in the far left corner of the experimental table.

Next, the inner brass container was removed with the magnet and set in its holder. The screw top was removed with the manipulators and set beside the castle lid in the far left corner of the table. The inner aluminum capsule was then dumped out of the brass container onto the table. The now empty brass container was returned to its holder.

The aluminum container was then picked up with the manipulator and placed in its holder located in the centre of the table in front of the castle. The top of this vial was unscrewed and set near the other lids. The open vial was then picked up with the manipulator and the six iridium pellets poured out onto the table in the pellet-handling area. The vial was then returned to its holder.

At this point the plastic fingers covering the manipulators were removed to present a clean surface for handling the pellet baskets and test tubes. This phase was completed at 1500 hours.

Using the suction probe, the pellets were then placed, one by one, in their ladles and thence into test tubes of group A in heater number 1. The order of loading was numbers 6, 5, 4, 3, 2, and 1. This phase was completed at 1510 hours.

C.2 Seven Day "Soaking" Period

During the next seven days, the pellets sat immersed in the water of test tubes A1 through A6. During this period there were no heaters required so temperature readings were only checked once a day. The results of these temperature readings are given in the first part of Table I. The relationship between the reading recorded and the actual temperature in ^{o}C was determined by Monserco and thus is not given here. The location of the ladles and their test tubes in this stage are shown in Figure 1.

C. TEST RUN (continued)

C.3 High Temperature Phase for Group A

On instructions from Monserco, the heater for Unit Number 1 was turned on at 1220 hours on April 8, 1981. The target thermocouple reading was said to be 62. According to Monserco, this represented the 50°C bath temperature required. Temperature readings were taken at least once an hour as given in Table I. the 50°C target was reached about 1300 hours. This phase was thus terminated at 1700 hours.

It was noted that the group A tubes had noticeably darkened.

C.4 Seven Day Humidity Stage (Group B)

At 1700 hours on April 8, 1981 the Unit 1 heater was turned off and the ladles, with their pellets, were moved from the Group A test tubes to those of Group B. This was accomplished, one by one, using the manipulators. As each ladle was about to be withdrawn from its Group A tube, it was shaken lightly to remove as much water as possible from the ladle. The ladle was then inserted into its Group B tube and checked to see that it was not immersed. These Group B tubes were already located in the heated block of heater Unit 2.

The transfers were performed in the following order: #1 ladle moved from test tube A1 to B1; #2 ladle from A2 to B2; #3 from A3 to B3; #4 from A4 to B4; #5 from A5 to B5; and #6 from A6 to B6. This phase was completed by 1705 hours when the Unit 2 heater was turned on.

The Group A test tubes were now moved, one by one, from heater Unit 1 to the rear test tube storage rack as shown in Figure 2. This was completed by 1710 hours. The test tubes from Group C were then moved from their storage locations in the front test tube storage rack to heater Unit 1 as shown in Figure 2. These tubes have had rubber stoppers in them since the beginning of the test. One of these, from C6, was dislodged during the transfer operation. It was replaced with a stopper from one of the spare tubes. These moves were completed by 1715 hours. Figure 2 reflects the location of the various test tubes at this point.

For the next seven days temperature readings were recorded at least twice a day as given in Table I. The target reading for heater Unit 2 was given as 42. By the end of this phase, it was noted that the test tubes of Group B had darkened.

C. TEST RUN (continued)

C.5 Final High Temperature Phase (Group C)

At 0756 on April 16, 1981 heater Unit 1 was again turned on in preparation for the final high temperature phase of the test run.

At 0910 hours heater Unit 1 read 62, the target reading. The rubber stoppers were removed from the Group C tubes and stored temporarily on a "clean" area of the table.

At 0914 hours the ladles and their pellets were transferred, one by one, from their Group B tube to the corresponding group C tube in heater Unit 1 in the following order: 1, 2, 3, 4, 5 and 6. The same procedure mentioned in Section C.4 was used. A visual check was made to ensure that all pellets were suitably immersed in their Group C tubes. These moves were completed at 0917 hours.

The two stoppers removed from tubes C5 and C6 were put on tubes A1 and A2. The other four stoppers from the Group C tubes were put on tubes B1, B3, B5 and B6.

Temperature checks were made at least once an hour, as given in Table I, until the test was terminated at 1324 hours, April 16, 1981.

C.6 Return of Pellets to Castle

The pellets were returned to their inner aluminum container, one by one, commencing with number 1 at 1325 hours. Some difficulty was encountered in removing the pellets from their baskets. Whether this sticking was due to retained moisture or an actual physical adhesion has not been determined. Despite holding each ladle against the blotting paper upon removal from its Group C test tube, the pellets still had to be prodded out of their ladles using the metal hook meant for handling the brass container. As each pellet was liberated from its ladle onto the table, it was transferred to the aluminum container via the suction tool and a plastic funnel. The handling order and times of transfer to the aluminum container are given below:

Handling order	Pellet No.	Time
1	1	1325
2	3	1330
3	2	1339
4	4	1340
5	5	1344
6	6	1347

With the plastic funnel removed and visually checked for any sticking pellets, the top of the aluminum container was screwed on at 1355 hours. The aluminum container was then reinserted into the brass container. By 1400 hours the top was secured on the brass container using the manipulators. The brass container was then transferred to the castle using the manipulators and the metal hook meant for the task. This was completed by 1402 hours and by 1404 hours, the castle lid was replaced using the remote block and tackle guided by the manipulators.

D. POST RUN CLEAN-UP

Following the above operations, the Hot Cell was opened and entry gained. There were no high radiation levels but contamination swipes of all apparatus and general working areas were taken and counted before proceeding. The results of these swipes are given in Table II.

After determining that the exterior of the castle was within acceptable contamination limits, the top was bolted on and the castle returned to its F113 shipping container. This operation was completed and F113-20 closed by 1440 hours.

As expected, the Mactac covering the manipulator pads was contaminated (see Table II). The protective Mactac "skin" was then removed and the manipulators reswiped and found free of contamination.

The end of the suction tool measured ~ 5 mr/hr at contact. The tubing leading to the suction tool was cut and the separated apparatus placed in a tin can along with the contaminated magnet and the eyelet used to hold the suction tool.

The pellet ladles measured about 10 mr/hr at contact and appeared to have a brownish stain where the pellets rested in them. These ladles were put in a separate tin can along with the plastic funnel for later counting.

Clean rubber stoppers were placed in all tubes not presently containing stoppers. Then group B and C tubes were transferred from the heaters to the test-tube racks for later counting. All apparatus and samples were left in the Hot Cell over the Easter weekend.

On Monday April 20, the test tubes and pellet ladles were removed from the Hot Cell. The apparatus was dismantled and removed from the Hot Cell. The contaminated blotter was carefully removed and disposed of.

A further check of the white table indicated some contamination in the right rear area where the empty ladles had been stored after retrieving the pellets. This was cleaned up.

E. DETERMINATION OF ACTIVITIES IN TEST TUBES AND LADLES

E.1 Calibration and Efficiency Determinations

The test run samples and the pellet ladles were counted using an APTEC Phyge Spectrometer Model CS8-D15C and a Tracor Northern Pulse Height Analyzer Model TN-1705. Two counting geometries were used, one at 30 cm to eliminate standard and sample geometry differences and one at 5 cm to get reasonable statistics for comparative measurements of the test tubes.

The Standard Source used was an Amersham multi-element standard #RO/130/1 which is traceable and certified. The peaks used and their resulting efficiencies are given in Table III.

Table III is relatively self-explanatory with the top section presenting the measured data and the lower section the resulting calculations. The efficiency figures calculated are counts per gamma-ray at the 30 cm distance. A plot of this is presented in Figure 3. The count interval was 3000 seconds with the source present and then a further 3000 seconds to subtract background.

E.2 Absolute Activity Determination of Test Tube A5

Following the calibration and efficiency determinations, an absolute determination of the activity in test tube A-5 was made with the tube placed 30 cm from the detector. The tube was counted for 300 seconds and the four main peaks analyzed as per Table IV. The three most prominent peaks were then used to calculate the absolute activity using interpolated efficiency data from Figure 3. The resulting average value was 12.6 μCi.

E.3 Determination of Activities in Test Tubes

The test tubes, containing the water samples, were counted 5 cm from the detector for 30 seconds to achieve reasonable statistics in a short time span. Since all tube geometries for Groups A and C, the most important ones, are identical, relative measurements could confidently be made in this manner. The only difference in Group B was less water in the test tubes. Thus, by counting all test tubes in a relative fashion and knowing the absolute activity contained in one of them (tube A5), the activities contained in each one could be calculated. From section E.2 above, tube A5 was known to contain 12.6 μCi of ^{192}Ir. Thus, by comparing the other tubes to A5, their contents were calculated. The results are given in Table V.

To gather more statistics a wide band around the 300 kev region was used to sum data over three major peaks (296, 308 and 317 kev). As each spectrum was collected, it was visually scanned for impurity peaks. None were found.

Two of the blank test tubes were also counted to check for contamination. As shown in Table V, they were at background levels.

E.4 Determination of Activities

The six pellet ladles were also counted to determine the activity remaining on them. They were counted for 100 seconds at 30 cm with the results as shown in Table VI. For these determinations, the isolated 468 kev peak was used with a detection efficiency of 2.14×10^{-4} c/γ and an absolute abundance of 49.5%.

Table VI contains both a Net Peak and a Net Count Column. From the background determinations given in the table, it was determined that the presence of the other test tubes in the room, even though more than ten feet away, gave rise to about 35 counts in the 468 kev peak area of interest. This value, which is totally within the peak area, and could easily be seen as a peak, was thus subtracted from each net peak area to get a true count for the sample being measured.

E.5 Impurity Check

The six test tubes from Group A, the ones most likely to contain contamination other than ^{192}Ir, were measured as a block in a 100 second count at 30 cm. The peaks found are given in Table VII. They are all ^{192}Ir peaks except for the Platinum X-rays arising from the decay daughter of ^{192}Ir and the lead X-rays arising from shielding around the detector. The source for checking these peaks was reference 1, part of which is reproduced in Table VIII.

REFERENCES

1. "Gamma Ray Energy Tables for Neutron Activation Analysis", R. H. Filby et al., Washington State University, 1970, Report WSUNRC-97(2).

TABLE I

TEMPERATURE LOG °C

DATE	TIME	UNIT #1	UNIT #2	REMARKS
April 1/81	1515	34	34	HOT CELL LIGHTS ON
" 2/81	1500	32	31	" " " OFF
" 3/81	1030	31	32	" " " ON
" 5/81	1430	31	30	" " " OFF
" 6/81	1100	31	30	
" 7/81	1400	31	30	
" 8/81	1000	33	32	HOT CELL LIGHTS ON OVERNIG
" "	1220	33	32	HEATER #1 ON
" "	1235	56	32	
" "	1317	61	32	
" "	1404	63	33	
" "	1450	63	33	
" "	1530	62	33	
" "	1612	63	33	
" "	1631	63	33	
" "	1700	62	34	HEATER #1 OFF
				PELLETS MOVED FROM UNIT #1
				TO UNIT #2
April 8/81	1705	58	34	HEATER #2 ON
" "	1716	46	40	
" "	1850	34	43	
" "	1950	33	42	
April 9/81	0850	33	41	
" "	1900	33	42	
April 10/81	0900	31	41	
" "	1900	31	42	
April 11/81	0035	31	40	
" "	1745	32	43	
April 12/81	1330	32	42	
" "	2040	32	42	
April 13/81	0845	32	42	
" "	1650	32	43	
April 14/81	1000	32	42	
" "	1900	32	42	
" 15/81	0940	31	42	
" "	1730	31	43	
April 16/81	0756	31	43	
" "	0910	62	44	HEATER #1 ON
				HOT CELL LIGHTS ON
				PELLETS MOVED FROM #2 to #1
April 16/81	0920	62	43	
" "	1017	62	42	
" "	1112	62	41	
" "	1203	62	43	
" "	1324	62	41	HEATER #1 OFF
				HEATER #2 OFF
				TEST TERMINATED

Humidity Chamber Temp. 30-33°C

Water Temp 50-51°C

Water Temp 50°C

TABLE II

POST RUN CONTAMINATION RESULTS

	AREA SWIPED	RESULT (dpm)	REMARKS
1.	Top of Lid of Castle	Bgd	
2.	Lower outside of castle	145	
3.	Outside of Castle	Bgd	
4.	White platform (table)	133	
5.	Inside box (clean area)	Bgd	
6.	Manipulators	10,822	Mac Tac still on
7.	Black table top	166	
8.	Heater Unit 1	374	
9.	" " 2	101	
10.	Test Tube Rack (Front	50	
11.	" " " (Rear)	67	
12.	One of Test Tubes (Group A)	62	
13.	Blotter surface	80,588	(Area where pellets were handled)
14.	Magnet	1112	Used to handle brass container
15.	Eyelet holding suction tool	5493	
16.	Manipulators (Right)	89	Mactac removed
17.	" (Left)	68	Mactac removed
18.	Shipping Container (side)	89	
19.	" " (top)	177	
20.	Box-centre	316	After blotter removed
21.	" -sides	85	

TABLE III

EFFICIENCY DETERMINATION

ISOTOPE	PEAK ENERGY	PK.CHAN.	INTEGRATION (No. of Chan)	GROSS AREA	BACKGROUND (Value Used)	NET AREA
Cd-109	88	115	112-117(6)	5070	200-191(196)	3894
Co-57	122	167	165-169(5)	3687	162-175(168)	2847
Ce-139	166	234.2	231-237(7)	2493	179-151(165)	1338
Sn-113	392	581	579-584(6)	1666	83-77(80)	1186
Sr-85	514	769	767-773(7)	755	63-49(56)	363
Cs-137	662	995.7	989-999(11)	6047	41-41 (41)	5596

SOURCE: AMERSHAM #RO/131/1 - REF. 1200 GMT 1-JUNE - 80

COUNT : 3000 SECONDS WITH SOURCE AT 30 cm

PEAK (kev)	γ's/sec AT CALIBRATION	HALF LIFE	DELAY FACTOR (1200 21-APR-81	CORRECTED γ/s	EFFICIENCY
88	3442	453D	.608	2093	6.20×10^{-4}
122	3082	270.9D	.435	1342	7.07×10^{-4}
166	3644	137.5D	.194	708	6.30×10^{-4}
392	10640	115.0D	.141	1500	2.64×10^{-4}
514	20309	64.8D	.0015	628	1.93×10^{-4}
662	12921	30.17Y	.980	12659	1.47×10^{-4}

* EFFICIENCY = (NET AREA/COUNT TIME)/(CORRECTED γ's/sec)

TABLE IV

ABSOLUTE DETERMINATION OF TEST TUBE A5

PEAK	CHANNELS	PEAK AREA†	BACKGROUND	NET AREA	EFFICIENCY*
296	430-438	18149	336-335	15125	3.65×10^{-4}
308	450-457	17057	302-209	15009	3.48×10^{-4}
316	460-470	42340	212-112	40558	3.38×10^{-4}
468	693-702	15132	50-27	14747	2.14×10^{-4}

† 300 SECOND COUNT AT 30 cm

* TAKEN FROM FIGURE 3

PEAK	γ's/sec†	ABSOLUTE** ABUNDANCE	REPORTED ABUNDANCE (REF 1)	μCi††	Average*** Activity
296	138128	29.6	27.4	13.6	
308	143764	30.8	30.5	12.7	12.6
316	399980	85.8	85.8	12.6	
468	229704	49.2	49.5	12.5	(0.47 MBq)

† γ's/sec = (NET AREA/COUNT TIME) /EFFICIENCY

** Based on 316 kev peak at 85.8% abundance (REF 1)

†† (γ's/sec)/(REPORTED ABUNDANCE)/(3.7×10^4 Bq. /μCi)

***The absolute and reported abundances of the 296 kev peak did not match as they did in the other peaks. An intercomparison of tables of radionuclides revealed inconsistences on the reported abundances for the 296 kev peak. Hence, this peak was not included in the absolute determination. It should be noted that had it been included, the average number would have been 12.8 instead of 12.6, i.e. a negligible variation of about two percent.

TABLE V

TEST TUBE ACTIVITY

t-t	Σ^*	Net Σ^+	Activity (μCi)	(MBq)
Background	150	-		
"	145	-		
C4	80,358	83,402	5.4	0.20
B2	877	730	0.05	1.85×10^{-3}
C1	48,511	48,364	3.1	0.11
C2	20,849	20,702	1.3	0.05
C3	25,109	24,962	1.6	0.06
C5	93,691	93,544	6.0	0.22
C6	22,801	22,654	1.4	0.05
A1	83,549	83,402	5.4	0.20
A2	36,419	36,272	2.3	0.09
A3	46,151	46,004	3.0	0.11
A4	123,786	123,639	7.9	0.29
A5	196,557	196,410	12.6 **	0.47
A6	49,346	49,199	3.2	0.12
B1	1,438	1,291	0.08	2.96×10^{-3}
B2	833	686	0.04	1.48×10^{-3}
B3	576	329	0.02	7.40×10^{-4}
B4	932	785	0.05	1.85×10^{-3}
B5	302	155	0.01	3.70×10^{-4}
B6	370	123	0.008	2.96×10^{-4}
Blank	147	-		
Striped Blank	125	-		

* Integrated over three large peaks (296, 308 and 317 kev)

\+ Background = (150 + 145)/2 = 147

** All results are based on A5 having 12.6 μCi numbers determined by efficiency calculation

TABLE VI

LADLE ACTIVITY

LADLE #	Σ+	BED (10 CHAN.)	NET PEAK	NET COUNT*	γ/sec+	μCi‡
1	2715	8-3 (6)	2655	2620	122,430	6.7
2	1054	4-2 (3)	1024	989	46,215	2.5
3	4112	11-7 (9)	4022	3987	186,308	10.2
4	5072	14-8 (11)	4962	4927	230,234	12.6
5	9923	32-15 (24)	9648	9648	450,841	24.6
6	2208	8-4 (6)	2113	2113	98,738	5.4
Background	35	(with beaker)				
Background	34	(w/o beaker)				
Funnel	61			27		

\+ 100 second count at 12" using 468 kev peak (Chan. 693-702)

* Net peak area - room background (35)

\+ γ/sec. = net count/count time/efficiency

‡ μCi = (γ/sec)/(.495γ/dis.)/(3.7 x 10^4 Bq./μCi)

TABLE VII

IMPURITY CHECK*

PEAK #	CHAN. #	ENERGY	NUCLIDE
1	81	66	Pt X-ray
2	94	74	Pb X-ray
3	109	84	Pb X-ray
4	288	201.1	^{192}Ir
5	295	205.7	"
6	433	295.6	"
7	452	308.0	"
8	465	316.5	"
9	554	374.5	"
10	618	416.2	"
11	698	468.4	"
12	723	484.6	"
13	883	588.9	"
14	907	604.6	"
15	919	612.4	"

*Gross count of Group A test tubes - 100 seconds at 12"

TABLE VIII

^{192}Ir PEAKS FROM REF. 1

ENERGY (KEV)	NUCLIDE	D	F	HALF-LIFE	R-I	A-I
201.3	77-IR-192	S		7.42E+01D	.6	.480
205.7		S			3.8	3.260
281.5		R			.3	.280
283.4		S			.4	.340
295.9		R			31.9	27.400
308.4		R			35.5	30.500
316.5		R			100.0	85.800
374.4		S			.7	.630
416.4		R			.9	.780
468.1		R			57.7	49.500
484.7		S			4.3	3.650
489.1		S			.4	.340
588.6		R			5.3	4.580
604.4		R			9.7	8.320
612.4		R			5.8	5.010
884.5		R			.3	.280
1063.0		R			.1	.050

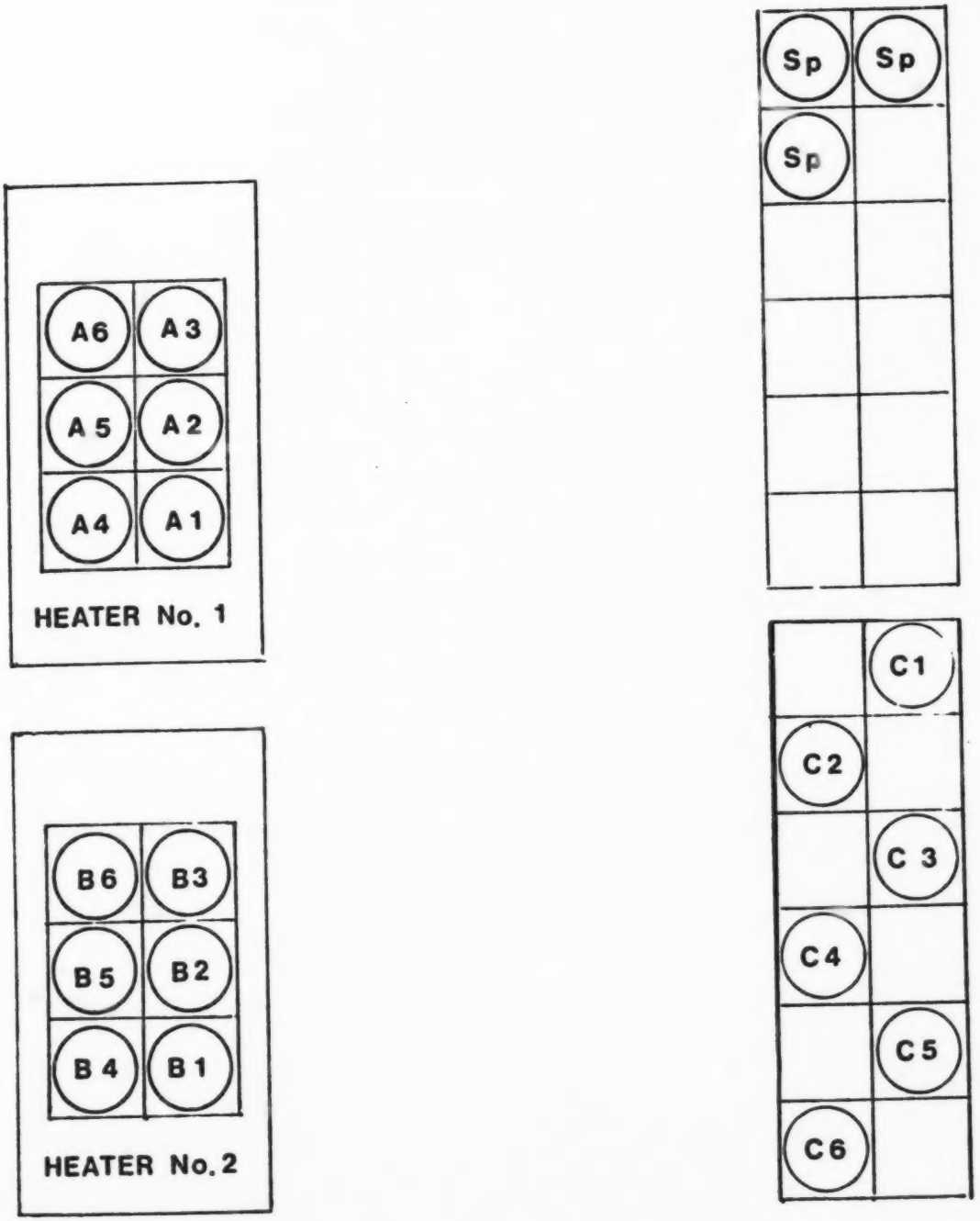

FIGURE 1. TEST TUBE LOCATIONS FOR FIRST HALF OF TEST

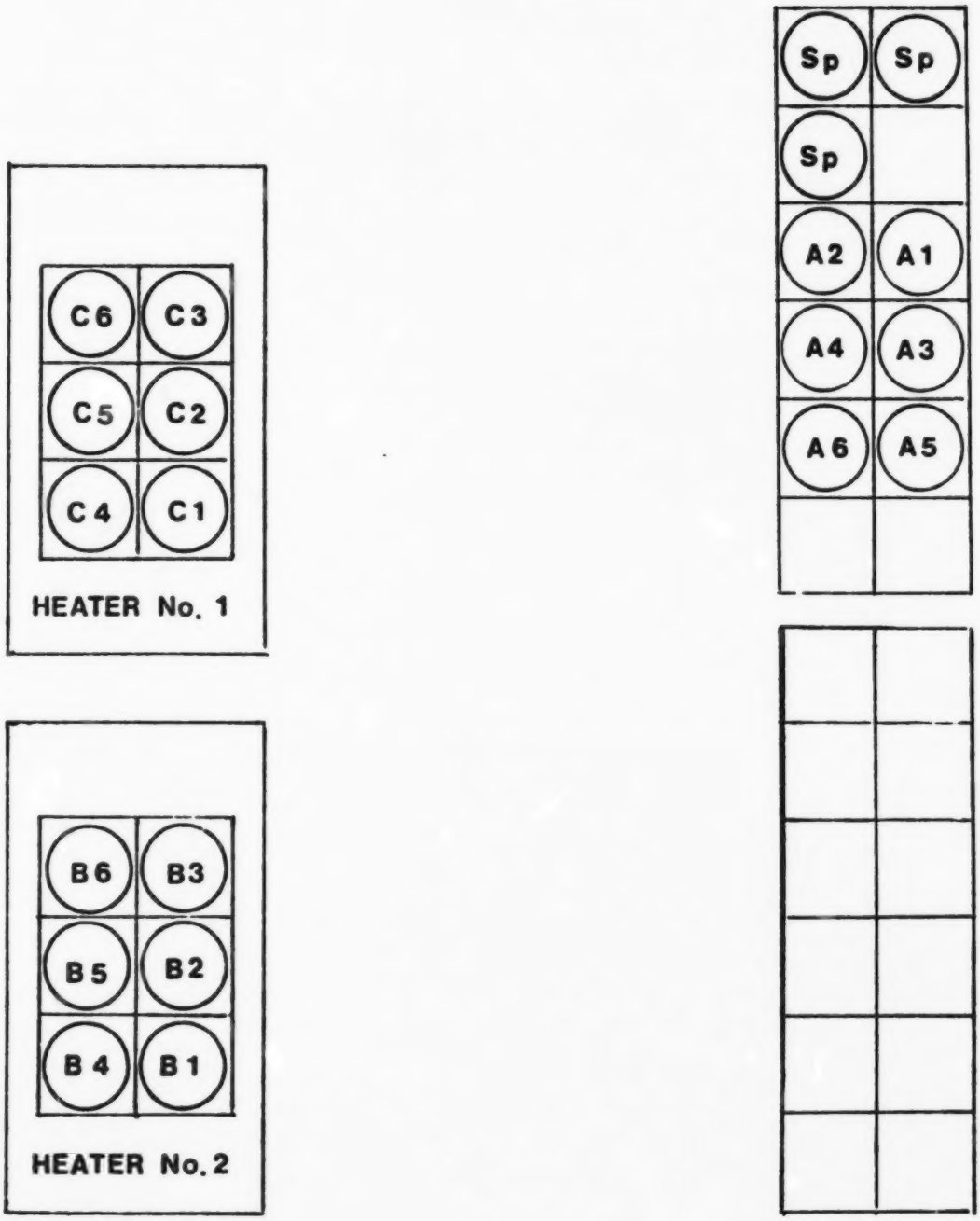

FIGURE 2. TEST TUBE LOCATIONS FOR LAST HALF OF TEST

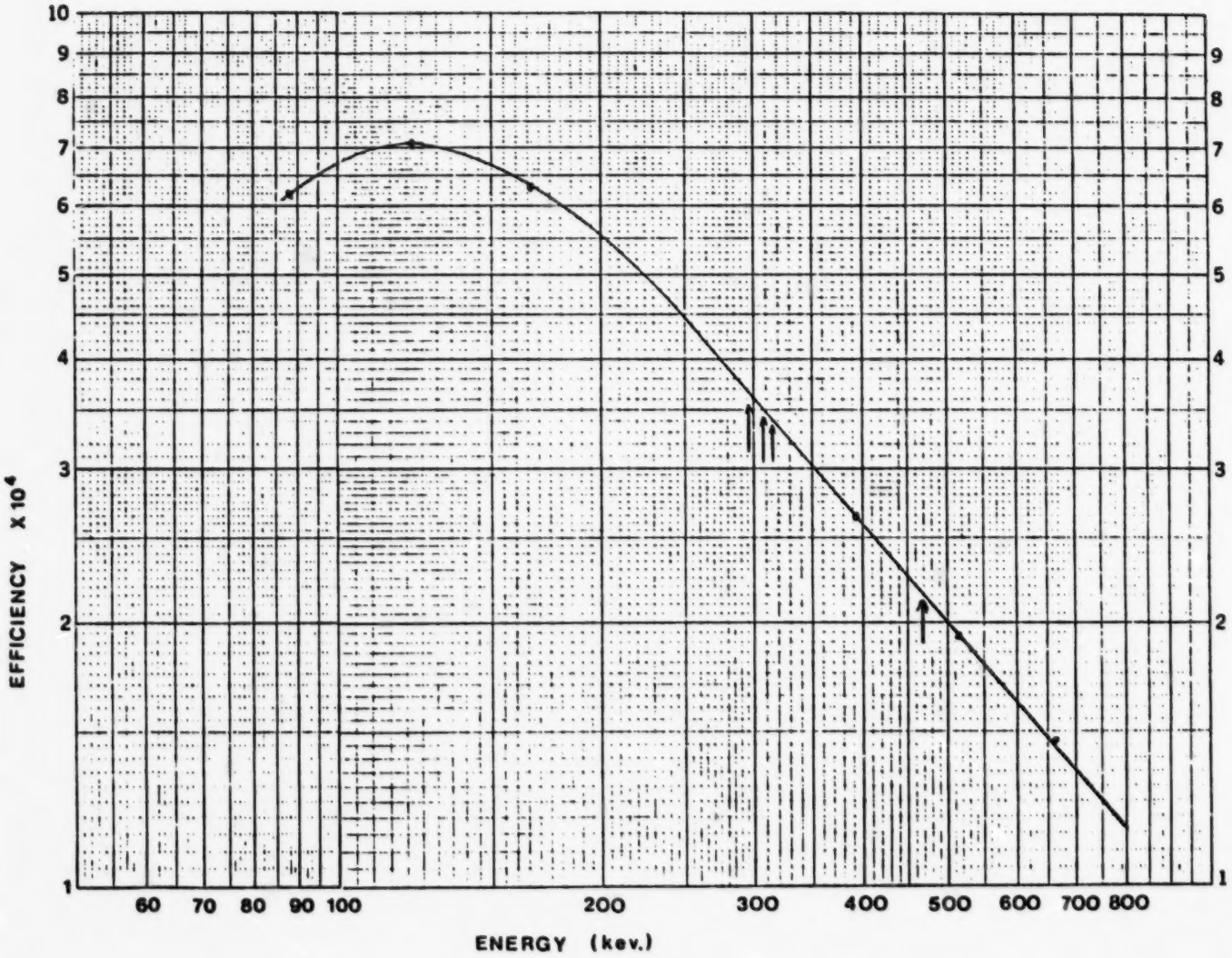

FIGURE 3. EFFICIENCY PLOT FOR DETECTOR

APPENDIX 2

IRIDIUM PROPERTIES BIBLIOGRAPHY

ASME Metals Handbook, Vol. 2, Ninth Edition

Mechanical Properties, Vol. 4. Edited by F. H. Wohlbier Trans. Tech. Pub. 1976

Selected values of Thermodynamic Properties of the Elements. Prepared by R. Hultgren et al. American Society for Metals 1973

Materials Handbook, Brady. Tenth Edition, McGraw Hill

New Engineering Materials: A conference arranged by the Process Engineering Group, 13th, 14th October 1965, Birmingham, U.K.

APPENDIX 3

TESTS FOR SPECIAL

FORM MATERIAL

Special form radioactive material

135. Special form radioactive material shall mean either an indispersible solid radioactive material or a sealed capsule containing radioactive material. The sealed capsule shall be so constructed that it can be opened only by destroying the capsule. Special form radioactive material shall meet the following requirements:

(a) It shall have at least one dimension not less than 5 mm

(b) It shall comply with the relevant test requirements specified in Section VII, paras 726-737.

The special form concept permits the inclusion of a greater activity in a Type A package.

TESTS FOR SPECIAL FORM RADIOACTIVE MATERIAL

General

726. The tests are: the impact test, the percussion test, the bending test and the heat test.

727. Specimens (solid radioactive material or capsules) to be tested shall be prepared as normally presented for transport. The radioactive material shall be duplicated as closely as practicable.

728. A different specimen may be used for each of the tests.

729. The specimen shall not break or shatter when subjected to the impact, percussion or bending tests.

730. The specimen shall not melt or disperse when subjected to the heat test.

731. After each test, a leaching assessment shall be performed on the specimen by a method no less sensitive than the methods given in paras 736 and 737.

Test methods

732. Impact test: the specimen shall fall onto the target from a height of 9 m. The target shall be as defined in para.708.

733. Percussion test: the specimen shall be placed on a sheet of lead which is supported by a smooth solid surface and struck by the flat face of a steel billet so as to produce an impact equivalent to that resulting from a free fall of 1.4 kg through 1 m. The flat face of the billet shall be 25 mm in diameter with the edges rounded off to a radius of 3 mm ± 0.3 mm. The lead, of hardness number 3.5 to 4.5 on the Vickers scale and not more than 25 mm thick, shall cover an area greater than that covered by the specimen. A fresh surface of lead shall be used for each impact. The billet shall strike the specimen so as to cause maximum damage.

734. Bending test: the test is applicable only to long, slender sources with both a minimum length of 10 cm and a length to minimum width ratio of not less than 10. The specimen shall be rigidly clamped in a horizontal position so that one half of its length protrudes from the face of the clamp. The orientation of the specimen shall be such that the specimen will suffer maximum damage when its free end is struck by the flat face of a steel billet.

The billet shall strike the specimen so as to produce an impact equivalent to that resulting from a free vertical fall of 1.4 kg through 1 m. The flat face of the billet shall be 25 mm in diameter with the edges rounded off to a radius of 3 mm ± 0.3 mm.

735. Heat test: the specimen shall be heated in air to a temperature of 800°C and held at that temperature for a period of 10 min and shall then be allowed to cool.

Leaching assessment methods

736. For indispersible solid material:

(a) The specimen shall be immersed for 7 days in water at ambient temperature. The water shall have a pH of 6 - 8 and a maximum conductivity of 10 μmho/cm at 20°C.

(b) The water with specimen shall then be heated to a temperature of 50° ± 5°C and maintained at this temperature for 4 hours.

(c) The activity of the water shall then be determined.

(d) The specimen shall then be stored for at least 7 days in still air of humidity not less than 90% at 30°C.

(e) The specimen shall then be immersed in water of the same specification as in (a) above and the water with specimen heated to 50° ± 5°C and maintained at this temperature for 4 hours.

(f) The activity of the water shall then be determined.

The activities determined in (c) and (f) above shall not exceed 0.05 μCi.

737. For encapsulated material:

(a) The specimen shall be immersed in water at ambient temperature. The water shall have a pH of 6 - 8 with a maximum conductivity of 10 μmho/cm. The water and specimen shall be heated to a temperature of 50° ± 5°C and maintained at this temperature for 4 hours.

(b) The activity of the water shall then be determined.

(c) The specimen shall then be stored for at least 7 days in still air at a temperature not less than 30°C.

(d) Repeat (a).

(e) The activity of the water shall then be determined.

The activities determined in (b) and (e) above shall not exceed 0.05 μCi.

APPENDIX 4

EQUIPMENT DESCRIPTION

1. Heater #1
   Lab-line Instruments Inc. Ill. U.S.A.
   Multilelock Heater No. 2090
   Temp. range: ambient tjo 130°C±0.5°C

2. Heater #2
   Thermo line Corp. Iowg, U.S.A.
   Model No. DB-17615
   Series No. 176
   Serial No. 2726
   Temp. range: ambient to about 130°C±0.5±C

3. Mercury Thermometer
   ERTCO (USA)
   Model No. B-93544
   Cert. (ASTM-15C)

4. Thermo couple instrumentation
   Thermoelectric thermocouple meter, -150 to 400°C
   Model 31630-00-013
   Thermocouples, Thermoelectric 1T1210A

5. See Appendix 1 for the activity, conductivity and pH equipment